Standard Operating Procedure for The Extraction of Polychlorinated Biphenyls (PCBs) from Soils, Sediments, Wipes and other Solid Wastes for the TSCA Program (Soxhlet Extraction)

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1. Scope and Application
   1.1. This standard operating procedure is applicable to the extraction of polychlorinated biphenyls (PCBs) from soils, sediments, wipes, and other solid waste samples using Soxhlet extractors, prior to gas chromatographic analysis. The following compounds can be extracted using this method: Aroclor 1016, Aroclor 1221, Aroclor 1232, Aroclor 1242, Aroclor 1248, Aroclor 1254, Aroclor 1260, Aroclor 1262 and Aroclor 1268. This method will be followed for all solid samples collected under the TSCA program.
   1.2. 40 CFR 761 mandates the use of U.S. EPA SW-846 Methods 3500C - Organic Extraction and Sample Preparation (for generalized sample preparation guidance), Methods 3540C - Soxhlet Extraction, or 3550C - Ultrasonic Extraction (for extraction of PCBs from solids) and Method 8082 - Polychlorinated Biphenyls by Gas Chromatography (for analytical measurement of extracted PCBs). For the determinative procedure, refer to CRL SOP GC003. No deviations from EPA method 3540C were performed for this CRL SOP.
   1.3. This method describes the procedure for extracting PCBs using Soxhlet extractors. This extraction technique ensures intimate contact between the sample and the extraction solvent. It is applicable to certain TSCA concentration standards for regulation compliance, otherwise known as "Action Levels". The laboratory must be informed of the action levels relevant to the samples submitted for analysis. These action levels are to be used as guidance in the selection of sample preparation variables for the purpose of optimizing the accuracy of PCB measurements. The following table lists common action levels for different sample types.

TSCA PCB Action Levels:

Sample Type	Common Applicable PCB Action Levels	Other PCB Action Levels: Site-specific
Soils	1, 10, 25 & 50 ppm	100 ppm
Sediments	50 ppm	1 - 50 ppm
Wipes	10 µg/cm^2	100/cm^2

   1.4. PCBs are to be determined on a weight per weight basis, or for liquids, on a weight per volume (e.g. milligrams/liter) if the density of the liquid is also reported [40CFR761.1(b)(2)]. Total PCBs, by regulation, are to be reported on a dry weight basis for solid, or "non-liquid" samples [40CFR761.1(b)(4)(i)], and on a wet weight basis for "liquid" samples [40CFR761.1(b)(4)(ii)]. The "40CFR761.3 Definitions" section defines "liquid PCBs" as a homogeneous, flowable material containing PCBs

and no more than 0.5% by weight non-dissolved material. "Non-liquid PCBs" are defined as materials containing PCBs that by visual inspection do not flow at room temperature or when no liquid passes in 5 minutes, when either a 100 g or a 100 ml aliquot is placed in a paint filter (mesh# 60±5).

1.5. When PCBs are determined in multi-phase samples, the phases (non-liquid/liquid or liquid/liquid mixtures) shall be separated before analysis and PCBs determined in each phase, per Section 1.4 above [40CFR761.1(b)(4)(iii)]. The phase separation is not required if a non-dissolved, non-liquid phase is <0.5% by weight of a PCB material; in this case the sample can be tested as a single phase "liquid" sample. For disposal of multi-phase PCB material, the individual phase with the highest PCB concentration defines disposal requirement [40CFR761.1(b)(4)(iv)].

It is important for TSCA PCB inspectors to identify any multi-phase samples/sample types with the Action Levels in order for the laboratory to conduct the appropriate phase separation prior to sample preparation procedures. Likewise, when the PCB contamination is suspected to be very high, e.g. spills near PCB transformers, the laboratory should be informed so that additional measures to prevent laboratory contamination can be put into effect.

1.6. The extracts from this procedure are expected to require removal of PCB interferences using Florisil columns/cartridges and sulfuric acid. If sulfur is present, as evidenced by a huge peak early in the chromatogram, then the extract will have to undergo a sulfur cleanup procedure. Sulfur will mask the early eluting peaks of the Aroclor thereby preventing accurate identification and quantitation. There are three reagents that can be used for sulfur removal: mercury, copper powder, and tetrabutylammonium sulfite (TBA).

Refer to the following cleanup procedures:
CRL SOPs GC014 (Method 3620C-FLORISIL), GC016 (Method 3600C-ACID), and GC019 (Method 3660B-SULFUR).

2. Summary of Method

2.1. The soil/sediment sample is mixed with anhydrous sodium sulfate, placed between two plugs of glass wool, and extracted with 1:1 (v:v) acetone/hexane in a Soxhlet extractor. The extract is then concentrated and solvent exchanged to hexane.

2.2. Wipes are extracted in the same manner as soils, without the addition of sodium sulfate. Dry solid wastes (such as auto fluffs, wood chips, paper, steel cuttings, and

other unusual, non-routine matrices) should be either ground, cut, pulverized, or reduced in size through appropriate means prior to extraction.

3. Abbreviations and Definitions

TSCA - Toxic Substances Control Act
CRL - Chicago Regional Laboratory of Region 5, U.S. EPA
SDS – Safety Data Sheet
SOP - Standard Operating Procedure
LIMS - Laboratory Information Management System
MB - Method Blank - Laboratory Blank see section 1.2.17 (QMP)
LCS/LCS dup - Laboratory Control Sample/Duplicate
MS/MSD - Matrix Spike/Duplicate
QC - Quality Control
TCMX & DCB - Tetrachlorometaxylene and Decachlorobiphenyl. TCMX and DCB are used as an internal standard in the analysis of Polychlorinated Biphenyl and pesticides.
PCB - Polychlorinated Biphenyl

4. Health, Safety and Waste Handling
   4.1. Health and Safety
      4.1.1. Users of this method should operate a formal safety program. Perform this method according to CRL Chemical Hygiene Plan located on the CRL share drive (G:\drive).
      4.1.2. The toxicity or carcinogenicity of each reagent used in this procedure has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. Exposure to any of these chemicals should be reduced to the lowest possible level by whatever means available such as the use of hoods during preparation steps, use of protective gloves/clothing and safety glasses. Review Safety Data Sheets (SDS's) for specific physical and health hazards including appropriate personal protective gear (safety glasses, gloves, lab coats) to be used. SDS's may be accessed at www.sigmaaldrich.com.
      4.1.3. **"WARNING"** Polychlorinated biphenyl (PCB) standards should be handled with extreme caution. Preparation of stock, intermediate, and working standards should be carried out in a hood.
      4.1.4. **"WARNING"** Samples may contain high concentrations of volatile toxic compounds. Sample containers should be opened in a hood and handled with gloves to prevent exposure.
   4.2. Waste Handling

4.2.1. Liquid organic wastes such as waste solvents, extracts and expired standards that are less than 50 ppm PCBs are disposed of in green labeled 5-gallon plastic containers located in a lab fume hood designated as temporary holding area. Organic wastes that are more than 50 ppm PCBs are disposed off in purple labeled 5-gallon plastic containers. Solid, hazardous wastes are placed in properly labeled yellow bags.

4.2.2. Refer to the *CRL Safety, Health & Environmental Compliance Manual* for more information on waste reduction. Additional information on waste reduction can be found in the CRL Environmental Compliance Plan and additional information on hazardous waste spills can be found in the CRL Hazardous Materials/Hazardous Waste Contingency Plan.

4.2.3. Report all major spills.

5. Cautions and Interferences

5.1. Method interferences may be caused by contaminants in solvents, reagents, glassware and other sample processing hardware that lead to discrete artifacts or elevated baselines in gas chromatograms. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks.

5.1.1. Glassware must be scrupulously clean. See SOP GEN008 for the washing of laboratory glassware/hardware at the CRL. Rinse all glassware once with acetone and twice with hexane before use (10-20 ml each).

5.1.2. The use of high purity reagents and solvents will greatly minimize interference problems.

5.2. Interferences by phthalate esters can pose a major problem when using an electron capture detector. These compounds generally appear in the chromatogram as large late eluting peaks. Avoiding the use of common flexible plastics that contain varying amounts of phthalates will minimize this type of interference problems.

5.3. Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of interferences will vary. Cleanup procedures eliminate certain types of theses interferences.

6. Equipment and Supplies

6.1. Glassware – cleaning instruction refer to section 5.1.1

6.1.1. Soxhlet extraction apparatus

6.1.1.1. Soxhlet extractors – 40 mm ID

6.1.1.2. Condensers, Allihn

6.1.1.3. Boiling flask, flat- bottom, 250 – 300 ml capacity

6.1.2. Cylinder- graduated, Class A
6.1.3. Pipettes – Class A, volumetric
6.1.4. Pipettes – disposable
6.1.5. Crucibles or evaporating dish for weighing/ drying out samples
6.1.6. Volumetric flask – 10 ml, class A
6.1.7. Glass Test tubes-disposable, 10 ml, with caps
6.2. Glass wool
6.3. Boiling chips
6.4. Forceps/large tweezers
6.5. TURBO-VAP 500 Closed-cell concentrator (Zymark, Inc.) – for concentrating larger volumes, up to 300 ml, to as low as 1 ml
6.6. TURBO-VAP LV Evaporator, Nitrogen Evaporator (Zymark, Inc.) – for concentrating smaller volumes, < 10 ml, to as low as 1 mL; also for solvent exchange purposes; connected to a supply of high purity nitrogen
6.7. N-EVAP Analytical evaporator (Organomation Associates) – with temperature control; connected to supply of high purity nitrogen for solvent-exchange or for concentrating smaller volume sample extracts (<10ml)
6.8. Re-Certified Supportive Equipment
6.9. Analytical balance – capable of weighing to the nearest 0.01g – certified annually
6.10. Certified thermometer – certified annually

Note: Reference GEN026 for equipment and supplies ordering instructions

7. Reagents and Standards

All of the vendors and part numbers for the reagents and standards listed below are what the CRL currently uses as of the date of this SOP and are listed specifically to facilitate the ordering process. The mention of trade names or commercial products in this SOP is for illustrative purposes only and does not constitute an EPA endorsement or exclusive recommendation for use. Analysts are encouraged to seek out equivalent alternatives to the items listed below. These specific vendors and part numbers are provided examples and other materials of equivalent quality may be substituted without altering this SOP. Refer to CRL SOP GEN026 for instructions and analyst responsibilities when purchasing reagents and standards.

The described preparations of the spiking standards, calibration standards, and QC samples below are not absolute requirements. Rather, the prepared concentrations, spike volumes, and overall calibration range may be tailored to the needs of the project with

consideration given to any action limits or regulatory thresholds the samples are being analyzed against and the nature of the samples and any interference present therein.

NOTE: Solvents used for sample preparation must be tracked by a LIMS ID.

7.1. Solvents

7.1.1. Hexane – pesticide quality {Burdick & Jackson (B&J) or equivalent}

7.1.2. Acetone - (B&J or equivalent)

7.1.3. Hexane:Acetone, 1:1 v:v – mixture must be prepared by the analyst/tech assistant prior to extraction. Use class A graduated cylinder to measure solvents.

NOTE: The following brands names, suppliers, and part numbers are stated in this SOP for illustrative purposes. No endorsement is implied.

7.2. **Spiking Solutions**: Preparation of these solutions must be properly documented in the CRL Standards Logbook located next to the instruments and Laboratory Information Management System (LIMS). All intermediate and working spiking solutions must be labeled with the LIMS ID, expiration date, and preparer's initials. All pertinent information such as Supplier, Lot No., expiration date, date of preparation, how standard was prepared and preparer's name must be recorded into the CRL Standards Logbook. All standards must be properly sealed with Parafilm and refrigerated at 4 ± 2 °C. Only class A volumetric pipettes, volumetric flasks, syringes or micro dispensers must be used for measurements or dilutions.

Note Standard solutions, whether for calibration or spiking, must be verified to be free from interferences by injecting on the GC, prior to use.

7.2.1. PCB Spiking solution (Supelco or equivalent) - Ideally, the spiking compound should be the same Aroclor that is present in the sample, if known from historical data or from previous screening. If unknown, then Aroclor 1016/1260 mix will be used. The level of spiking should be consistent with the TSCA action level. Aroclor spiking solutions at 10, 50, 100, 200, and 500 µg/ml in acetone are the typical concentrations used. The final concentration of the Aroclor in the diluted extract should be near the midpoint of the five initial calibration levels.

7.2.2. Surrogate Spiking solution - consists of a mixture of Tetrachlorometaxylene (TCMX) and Decachlorobiphenyl (DCB) at 0.2 µg/ml of each component in

acetone; this is prepared by diluting a commercially available surrogate mix at a stock solution concentration of 200 µg/ml of each component with acetone (Supelco or equivalent). For action levels greater than 5 ppm, prepare a surrogate spiking solution at a higher concentration. The final concentration of the surrogates in the diluted extract should be near the midpoint of the 5 initial calibration levels.

Note: Addition of the spiking solutions to the samples/QC samples must be witnessed by another analyst & recorded on the bench sheet.

7.2.3. Expiration dates of primary standards (stock from vendor) are determined by vendor. Intermediate standards prepared in the laboratory expire one year from the date of preparation. Working spiking solutions expire six months from the date of preparation.

7.3. Sodium sulfate, granular, anhydrous, reagent grade. Purify by heating at 105 ºC for 12 hours or more, cool in a desiccator, and store in a glass bottle.

Note: Storing open containers of sodium sulfate in the laboratory may result in contamination. Cover the containers with aluminum foil.

7.4. Ottawa Sand – Fisher reagent or equivalent; contaminant-free

7.5. Method blank (MB): Use clean Ottawa sand (reagent grade) to prepare the method blank. The method blank should be carried through the entire procedure, including all the cleanup procedures, in order to verify that contamination has not occurred. A method blank must be run for every 20 samples, or less, if batch size is <20 samples; prepared at the same time, by the same procedure, and on each day of extraction. Add 1 ml of the surrogate spiking solution (0.2 µg/ml) to the method blank.

7.6. Laboratory Control Sample/ Laboratory control sample duplicate (LCS/LCSD): Add 1 ml of the PCB spiking solution (10 µg/ml) to the LCS/LCS duplicate (soil/sediment and wipes). The LCS/LCS duplicate extract final volume will be 10 ml.

7.7. Matrix Spike/Matrix spike duplicate (MS/MSD): Add the PCB spiking solution to the soil/sediment MS/MSD. The amount of spiking solution added must be consistent with the TSCA action level. The spiking solution used will be the same Aroclor type present in the sample, if known from site history or from previous screening. If unknown, Aroclor 1016/1260 will be used. (refer to 7.2.1)

8. Sample Handling and Preservation

8.1. The samples for this procedure are collected in clear glass bottles, approximately 8 oz. capacity, with Teflon-lined screw caps, and properly identified with sample tags. Sample collection is carried out according to a TSCA PCB Program QAPP.

8.2. The samples are delivered to the laboratory in coolers and until extraction should be refrigerated as stated in the sample handling SOP. (refer to GEN 013)

8.3. There is no extraction holding time requirement for sediment /soil and other solid samples submitted under the TSCA program. The extracts must be kept in tightly covered containers and stored in a samples-only refrigerator at 4 ± 2°C when not in use. The extracts must be analyzed within 40 days of extraction.

9. Sample Preparation and Analysis

9.1. LIMS: Batch/Bench sheet preparation

Create a batch and extraction bench sheet in LIMS prior to processing the samples. The bench sheet will contain the sample ID numbers and the QC samples included in the batch, the type and amount of spikes and surrogates added, and the sample source for the matrix spikes. Print a hard copy of the bench sheet and use it for recording the weights of the samples, which will then be entered later in LIMS and used in the calculations of the final results. The printed bench sheets will also include the name of the analyst/s who prepared the samples, the analyst who added the spikes and the analyst/tech support who witnessed the spiking.

9.2. Sample Handling

9.2.1. Soil/sediment sample – Mix the sample thoroughly, especially composite samples. Note the overall appearance of the sample, how much water or liquid phase is present; whether foreign objects such as sticks, leaves, rocks, etc., are present. **(NOTE: If the sample is multi-phase, consult the client on whether one or both phases need to be analyzed.)** Decant and discard any water layer if the client wants only the solid portion analyzed. Alternatively, if the client requires the analysis of both phases, then pour the liquid layer into a separate container, measure and conduct the appropriate extraction procedure. Prior to weighing, discard foreign objects, unless instructed otherwise by the client.

9.2.2. Wipes- The entire wipe sample is extracted. Rinse the sample container with the extraction solvent and add to the Soxhlet extractor.

9.2.3. Solid wastes – Dry, solid wastes (such as wood chips, paper, auto fluff, steel cuttings, and other unusual, non-routine matrices) should be either ground, cut, pulverized, or reduced in size through appropriate means prior to extraction.

9.2.4. Information about the action level should be obtained from the requestor, if not already specified in the chain of custody form.

9.3. Dry weight determination (% Total Solids):

NOTE: This does not apply to wipe samples and other wastes not amenable to drying.

Results should be reported on a dry weight basis, unless otherwise instructed by the client. Use the % solids to convert the wet weight of the sample extracted to the dry weight equivalent. The % solids determination is performed according to CRL SOP AIG019 or AIG020. The % solids are entered into LIMS to be later applied in the calculations on a dry weight basis.

9.4. Soxhlet Extraction

9.4.1. Prior to setting up the Soxhlet extraction apparatus, rinse the individual components (extractor, flat-bottom round flasks, & glass wool) with small amounts of extraction solvent (see 7.1.3).

NOTE: For wipe samples, omit steps 9.4.2 to 9.4.5 and proceed with the sample extraction as described in 9.4.6. Prepare the method blank and LCS/LCS duplicate using dry, clean, wipe material, such as gauze. There will be no MS/MSD samples for wipes.

9.4.2. Using a calibrated balance, weigh out a sufficient amount of the homogenized wet sample that would be equivalent to 10 - 15 grams of dry sample for the analysis. Immediately weigh out another 5 - 10 grams representative sample for the % solids determination (See Section 9.3). Repeat the procedure for all the samples.

9.4.3. Weigh out two additional aliquot portions of the sample designated for spiking and label as MS & MSD.

9.4.4. Weigh out triplicate 10 gram portions of Ottawa sand. Use one portion as the method blank (MB) and the other two portions as the LCS and LCS duplicate.

9.4.5. Blend the sample with an equal amount of sodium sulfate until the sample looks dry and powdery. Use more sodium sulfate if necessary.

9.4.6. Place the sample in between the plugs of glass wool in the Soxhlet extractor. Add the required amount of surrogate spike to each site sample and QC sample. The amount of surrogates added to the site samples will depend on the TSCA action level and appropriate adjustments are done if dilutions are anticipated, as

in the case of high levels of contamination. Add 1 ml of the surrogate spike (0.2 µg/ml) to the MB and LCS/LCS duplicate.

9.4.7. See section 7.7 for MS/MSD preparation.

9.4.8. See section 7.6 for LSC/LCSD preparation.

9.4.9. Turn on the heating block and cool water supply. Extract the sample using 250 - 300 ml of 1:1 acetone/hexane for 16 hours. Make sure the solvent is draining freely into the flask throughout the extraction process. At the end of 16 hours, turn off the heat; allow it to cool and concentrate and solvent exchange the extract using the following techniques.

9.5. Concentration:

9.5.1. Concentration using the TURBO-VAP 500 closed-cell concentrator

Description of the Apparatus:

The TURBO-VAP 500 closed-cell concentrator is a microprocessor-controlled concentrator that provides automated sample concentration using a helical gas flow. The closed cell consists of a motor assembly, a cell cap, condenser and concentrator tube. The cell cap, which is attached to the motor assembly, encloses the fan blade.

The concentrator unit consists primarily of a front panel for controlling the setup and operation, condenser holders and a water bath for warming the sample during concentration. It is equipped with quick-disconnect inlet/outlet for supplying coolant to the condensers and alarm volume control for adjusting the loudness of the buzzer that signals the end-point.

(NOTE: For a more detailed description of the apparatus and its operation, refer to TURBO-VAP 500 Closed Cell Concentrator Operator's Manual. A user's guide to daily operation of the instrument can be found in a plastic pocket at the side of the instrument.)

9.5.1.1. Fill the water bath such that when the sample tubes are placed in the water bath, the water does not overflow and the level of water in the bath is as high as the initial solvent in the sample tube.

9.5.1.2. Turn the unit **ON** and observe the "Power Up" diagnostics that occurs to make sure the instrument is functioning properly.

9.5.1.3. Recommended follows:

Water bath temperature – ranging from 45- 55°C

NOTE: Check the water bat h temperature with a certified thermometer. In the water bath temperatures logbook, record the evaporator ID, the setpoint temperature, the digital temperature readout on the device, and the certified thermometer ID and temperature.

Fan speed – 6000 rpm (Setting C)
Endpoint selection – Sensor endpoint

NOTE: Ideally, by selecting **SENSOR ENDPOINT**, concentration will proceed until the sensor detects a level of 0.5 or 1 ml depending upon the type of the concentrator tube. The fan speed may be changed if necessary *(This feature does not always work as it should; consequently, causing the extract to dry out. It is sometimes necessary to manually start and stop the* concentrators.)

9.5.1.4. Set the water bath temperature, fan speed and endpoint selection function specified above.

9.5.1.5. Make sure that the coolant supply and the solvent drain hose are properly connected. Check that the solvent waste container is empty. Pour the recovered solvent to a centralized waste collection container (see section 4.2).

9.5.1.6. Turn on the cold water supply (or water chiller, see instructions posted near equipment) connected to the condensers.

9.5.1.7. Rinse the TurboVAP 500 system prior to concentrating sample extracts in order to prevent cross contamination from previous extract, as follows: Pour 50 – 100 ml of acetone into the concentrator tube and place in the water bath. Grasp the motor assembly/cell cap and condenser as a unit, lift up from the mounting bracket and place on the concentrator tube. Start the concentration process by pressing the **START/STOP** button for the cell position used. Run for 5 – 10 minutes. Stop the system and discard any remaining acetone in the concentrator tube into the centralized waste collection container. Proceed to rinse tube with hexane (see section 5.1.1)

9.5.1.8. Transfer the sample extracts into the concentrator tubes; place the tubes in the water bath. Grasp the motor assembly/cell cap and condenser as a unit, lift up from the mounting bracket and place on the concentrator tube.

(Note: Cover any unused position with plastic closures supplied with the unit to minimize bath water evaporation.)

9.5.1.9. Start the concentration process by pressing the **START/STOP** button for each cell position used.

9.5.1.10. When a cell reaches its selected end point, its green **DONE** light will blink and the alarm sounds briefly at 30 second intervals. Silence the alarm by pressing the **START/STOP** button twice.

9.5.1.11. Dissemble the closed cell and **remove the sample tube promptly from the water bath to avoid further drying up of the extract**.

9.5.1.12. Return the motor/cell cap assembly to its mounting bracket position and the condenser to its holding cup.

9.5.1.13. Transfer the concentrated extract into a test tube. Proceed with the solvent exchange, using either 9.5.2 or 9.5.3 procedures.

9.5.1.14. Clean the glassware thoroughly for subsequent use.

9.5.1.15. Turn the unit off when no longer in use and replace the plastic closures in each cell position.

9.5.2. Concentration/Solvent Exchange Using the N-EVAP (Organomation Associates)

9.5.2.1. For small adjustments to the final volume, or to exchange solvent, concentrate the extract further using the N-EVAP (Organomation Associates, Inc.) This is done by placing the concentrator tubes in the N-EVAP at approximately 40 ºC and using a gentle stream of pure nitrogen applied to the sides of the tube, just above the surface of the extract.

9.5.2.2. Rinse down the internal walls of the tube several times with hexane during evaporation. Ensure that the solvent level in the tube is below the level of the water bath to prevent water from condensing into the sample. Do not reduce the extract volume to below 1 ml nor allow it to go to dryness.

9.5.2.3. To solvent exchange, reduce the volume of the extract to between 1 to 2 ml then bring the volume of the extract back up to at least 5 ml using hexane. Repeat this entire process at least two more times to ensure that the solvent has been sufficiently exchanged to hexane.

9.5.2.4. Make up the volume of the extract to 10 ml with hexane. Proceed with the required cleanup procedures.

9.5.3. Concentration/Solvent Exchange Using the TURBO-VAP LV (Zymark, Inc.)

(Note: Consult the Operating Manual for the proper operation of this unit. A user's guide to daily operation is located in a plastic pouch at the side of the instrument.)

The following brands names, suppliers, and part numbers are stated in this SOP for illustrative purposes. No endorsement is implied.

9.5.3.1. Turn ON unit and gas supply. Check the gas supply, gas pressure, water bath level and temperature.

9.5.3.2. Set the evaporation time with the TIME push wheel. The buzzer will sound when this time expires.

9.5.3.3. Load the 10 ml test tubes containing the extracts into the racks in the evaporator. The test tube must be positioned such that the nozzle supplying the gas for evaporation extends properly into it. There are five manifold rows with 10 nozzles each. Any or all of the rows may be selected by using the tube station pushbuttons. Place an empty test tube in any unused position in the row that contains the samples in order to minimize water bath evaporation.

9.5.3.4. Select the rows that contain sample tubes by pressing the corresponding TUBE STATION pushbuttons.

9.5.3.5. Press the START pushbutton. The unit will run for the duration of the set time. When the evaporation time expires, the buzzer will sound every 30 seconds and the gas will automatically shut off. Lift the cover, add more hexane (if exchanging solvent) and re-start the unit.

9.5.3.6. (NOTE: Do not let the solvent go dry. The optimal operating settings must be determined prior to using this unit by observing the time it takes to evaporate an amount of solvent, approximately equal to the amount of sample to be processed, down to the desired level. To ensure that all the samples will take about the same processing time, start with approximately equal amount of samples.)

9.5.3.7. To solvent exchange, reduce the volume of the extract to between 1 to 2 ml then bring the volume of the extract back up to at least 5 ml

using hexane. Repeat this entire process at least two more times to ensure that the solvent has been sufficiently exchanged to hexane.

Make up the volume of the extract to 10 ml with hexane in a volumetric flask. Proceed with the required cleanup procedures.

10. Quality Control

The laboratory is required to operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and an ongoing analysis of method blanks, spiked samples, laboratory control standards, and surrogate spikes to evaluate and document data quality. **For QC acceptance limits, refer to the determinative procedure** (CRL SOP GC003 - Standard Operating Procedure for the Determination of Polychlorinated Biphenyls by Gas Chromatography - Electron Capture Detection for TSCA Compliance).

10.1. QC for Soils/Sediments & Wipes

10.1.1. Method blank (MB): See section 7.5

10.1.2. Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSDUP): Prepare the LCS and LCS Duplicate by adding the PCB spike to two 10 gram (dry) aliquots of Ottawa sand (reagent grade). The LCSs should be run with every 20 samples, or less, if batch size is <20 samples; prepared at the same time, by the same procedure, and on each day of extraction. The LCS results are used to verify that the laboratory can perform the analysis in a clean matrix. This is especially very helpful when the sample matrix causes problems with spike recoveries. See Section 9.4.8 for the spiking procedure.

Use clean, dry wipe material (See 10.1.1) to prepare the wipe LCS/LCS duplicate.

10.1.3. Matrix spike/matrix spike duplicate (MS/MSD): The matrix spike and matrix spike duplicate are used to document the effect of the matrix on method performance. The MS/MSD should be run at a frequency of 1 for every 20 samples, or less, if batch size is <20 samples, and must be carried through the entire procedure, including cleanup procedures. Use the sample designated by the requestor/sampler; otherwise, choose a sample at random to be used as the MS/MSD. The sampler must provide an amount of sample designated as MS/MSD that will be sufficient for both the sample analysis and MS/MSD analysis. Spike the sample at a level consistent with the action level for the particular site or study. See Section 9.4.7 for the spiking procedure.

There are no MS/MSD wipe samples.

10.1.4. Surrogate spikes: Surrogate recovery data are used to evaluate data quality. Recoveries outside of the acceptance limits, assuming there are no errors in the calculations, amount spiked or concentrations of the spiking solution, may indicate analytical problems that must be investigated and corrected. If the presence of interferences after cleanup affects the calculations, it should be documented in the case narrative.

See 9.4.6 for the surrogate spiking procedure for the site samples.

10.2. QC Checks for other solid samples, since the matrix type cannot be known until the samples are received at the CRL, will depend on the type of matrix and will be determined on a case by case basis. The analyst should check with the GC Group Leader, Sample Coordinator, and the client prior to preparation.

11. Data and Records Management
    11.1. Raw data and bench sheets are to be submitted with the data package to the CRL data coordinator.
    11.2. Reagents and standards LIMS identification numbers used for procedure should be recorded on bench sheets and reviewed and submitted along with data package.
    11.3. All reviews are to be performed following the analytical procedure CRL.SOP GC010 and data review procedure CRL.SOP GEN 015.
    11.4. All electronic records associated with any data package generated must be archived following CRL.SOP GEN001.

12. Troubleshooting
    12.1. For a more detailed description of the apparatus and its operation, refer to TURBO-VAP 500 Closed Cell Concentrator Operator's Manual. A user's guide to daily operation of the instrument can be found in a plastic pocket at the side of the instrument
    12.2. Refer to Operating Manual for the proper operation of TURBO-VAP LV (Zymark, Inc.). A user's guide to daily operation is located in a plastic pouch at the side of the instrument.

13. Preventative Maintenance

13.1. Preventative maintenance records, user manuals and logbooks are kept with the instrument.

14. References

14.1. SW-846 Revision 2 November 1990 Method 3540C SOXHLET Extraction

14.2. Zymark TurboVap 500 Closed-Cell Concentrator Operator's Manual

14.3. Zymark TurboVap LV Evaporator Operator's Manual

15. Revision History

Version	Status*	Location of Change History
2	R	Section 4.2 included SDS information.
		Added revision table section 15.
1	R	1st version published to Qualtrax, same as GC014 Rev. 4.0 with minor formatting changes
		Entire document – Added header and adjusted spacing
		Title page – Deleted signature lines
6	R	Editing changes were made throughout the document to improve clarity
		Section 4.0 was combined with section 11.0
		SOP template was reformatted to be in compliance with new GEN006 template.
		Added "warning" to section 5.
		Added supportive re-certified equipment to section 6
		Defined MB,LCS,LCSD, MS,MSD in section 7
		Section 9.5.1.3 added note to reference thermometer log requirements.
		Section added Report all major spills and reference chemical hygiene plan in section 4.0.